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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SOLDERS (SOFT AND ROSIN-CORED)

PART 1 DETERMINATION OF TIN AND ANTIMONY

(First Revision)

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SOLDERS (SOFT AND ROSIN-CORED)

PART 1 DETERMINATION OF TIN AND ANTIMONY

(First Revision)

Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee, SMDC 34

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SOLDERS (SOFT AND ROSIN-CORED)

PART 1 DETERMINATION OF TIN AND ANTIMONY

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 1) (First Revision) was adopted by the Indian Standards Institution on 21 February 1983, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 21 grades of soft solder and 5 grades of rosin-cored solder have been specified in IS : 193-1982* and IS : 1921-1975† respectively. In order to determine correctly the composition of different solders, methods of analysis have been described in various parts of this standard. Due consideration has also been given in preparation of this standard to the facilities available in the country of such analysis.

0.2.1 This standard was first published in 1959. In this revision, the committee responsible for the preparation of this standard decided to publish this standard in four parts. In this revision, methods for determination of bismuth and silver have been included in Part 3 and Part 4 of the standard respectively. In this part and in Part 2 of the revised standard, preparation of sample for the analysis of rosin-cored solder covered in IS : 1921-1975† has also been included. This part covers determination of tin and antimony. The other parts cover the following:

Part 2	Determination of iron, copper and arsenic
Part 3	Determination of cadmium, zinc, aluminium, bismuth and nickel
Part 4	Determination of lead and silver.

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*Specification for soft solder (*fourth revision*).

†Specification for rosin-cored solder wire (*first revision*).

The method for determination of antimony stipulated in earlier edition has been substituted by potassium bromate titration method.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part 1) covers methods of chemical analysis of tin and antimony in lead and tin base solders (*see* IS : 193-1982† and IS : 1921-1975‡) having chemical composition within the following range. Lead shall be determined by difference except for Grade Sn95 Sb5 of IS : 193-1982† for which Part 4 of this standard may be followed:

Constituents	*Percent*
Tin	1-96
Antimony	0·05-6·0
Lead	Over 30

2. SAMPLING FOR CHEMICAL ANALYSIS

2.1 Samples shall be drawn in accordance with the procedure laid down in IS : 193-1982† and IS : 1921-1975‡.

2.2 Preparation of Sample (for Rosin-Cored Solder) — Samples shall be prepared as mentioned below:

a) From each reel or coil taken in accordance with **2.1**, cut a length of solder of mass approximately 50 g.

b) Melt the five lengths in a clean silica crucible just above the liquidus temperature of the alloy and mix thoroughly, using a stirrer that will not contaminate the melt. After the flux has floated to the top, carefully pour the alloy into a cool mould so as to form a bar approximately 6 mm thick that does not contain any flux. Clean the bar of flux residue by methanol washing and then thoroughly wiping dry.

c) Make cuts with a clean, fine saw across the entire width of the bar at regular intervals along its length. Do not use any lubricant during this operation. Treat the sawings carefully with a magnet to remove any particles of steel that may have been introduced during sawing. Mix the sawings thoroughly.

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*Rules for rounding off numerical values (*revised*).

†Specification for soft solder (*fourth revision*).

‡Specification for rosin-cored solder wire (*first revision*).

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals shall be employed in tests and distilled water (conforming to IS : 1070-1977*) shall be used where the use of water as a reagent is intended.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the result of analysis.

4. DETERMINATION OF TIN BY THE IODIMETRIC METHOD

4.1 Outline of the Method — The sample is decomposed by concentrated sulphuric acid and potassium bisulphate, and the tin in solution of the sample is reduced by measured amount of nickel. The tin in solution is then titrated with standard potassium iodate solution.

4.2 Apparatus

4.2.1 It shall consist of a 500-ml conical flask fitted with a three-hole rubber stopper containing an inlet tube for carbon dioxide and air condenser and a hole for a burette (*see* Fig. 1).

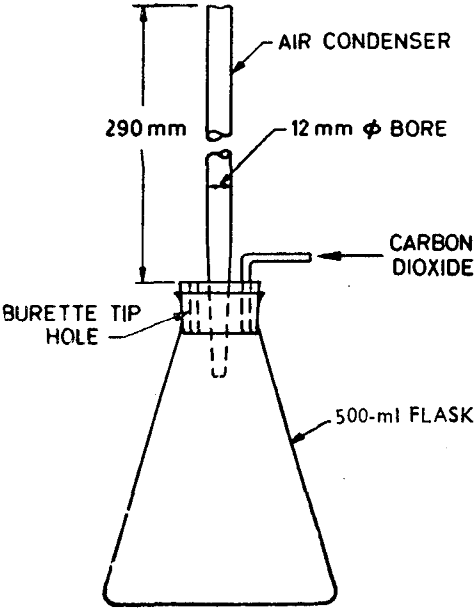

FIG. 1 APPARATUS FOR REDUCTION OF TIN

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*Specification for water for general laboratory use (*second revision*).

4.3 Reagents

4.3.1 *Concentrated Sulphuric Acid* — rd 1·84 conforming to IS : 266-1977*.

4.3.2 *Potassium Bisulphate* — solid.

4.3.3 *Concentrated Hydrochloric Acid* — rd 1·16 conforming to IS : 265-1976†.

4.3.4 *Nickel/Iron* — iron in the form of powder or nickel in the form of sheet.

4.3.5 *Potassium Chlorate* — solid.

4.3.6 *Starch Solution* — Make a suspension of 1 g of soluble starch in about 10 ml of water and add it carefully to 100 ml of boiling water. Boil for two to three minutes and cool. Prepare the solution fresh as needed.

4.3.7 *Carbon Dioxide* — oxygen-free gas.

4.3.8 *Standard Potassium Iodate Solution (0·1 N Approximately)* — Dissolve 3·567 g of recrystallized potassium iodate in 200 ml of water containing 1 g of sodium hydroxide and 10 g of potassium iodide. When the solution is complete, dilute to one litre with water in a volumetric flask. Standardize against an alloy of tin, antimony, lead and copper in approximately the similar proportion as present in the sample and reduce the tin and titrate with potassium iodate solution as described in **4.4**.

4.4 Procedure

4.4.1 Transfer a suitable mass of representative sample (so as to contain 0·10 to 0·30 g of tin) to a 500-ml conical flask. Add 20 ml of concentrated sulphuric acid and 5 g of potassium bisulphate, and heat to decompose the sample. Avoid very high temperature during the initial beating; otherwise sample may melt and an incomplete decomposition may result. When decomposition is complete, heat vigorously over an open flame to expel the sulphur on the walls of the flask and cool

4.4.2 Add 180 ml of water to the flask. Add 75 ml of concentrated hydrochloric acid and at least 5 g of roll of nickel sheet. Cover with a watch-glass, heat to boiling and boil gently for 30 minutes. Filter off nickel sheet, copper and antimony through a coarse filter paper or glass wool and collect the filtrate into a 500-ml conical flask and wash the filter with hot water. Preserve the filtrate.

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*Specification for sulphuric acid (*second revision*).

†Specification for hydrochloric acid (*second revision*).

4.4.3 Wash the precipitate of copper and antimony back into the original flask, add 10 ml of concentrated hydrochloric acid and a few crystals of potassium chlorate. Heat gently to effect complete dissolution of the metal. Dilute to 50 ml and boil to expel free chlorine. Add about 3 g of roll of nickel sheet or iron powder and boil gently for 15 minutes. Filter through a coarse paper or glass wool into the conical flask containing the filtrate preserved under **4.4.2**. To the filtrate, add 5 g of nickel sheet and fit up the apparatus as shown in Fig. 1. Heat to boiling with continuous evolution of gas for about 30 minutes. Some undissolved nickel should remain after reduction. (When antimony and copper content is not appreciable, repeated reduction of tin by nickel is not necessary.)

4.4.4 After the reduction is complete, cool the solution in the flask to about 10°C maintaining an atmosphere of carbon dioxide. Add 5 ml of starch solution and titrate with standard potassium iodate solution.

4.5 Calculation

$$\text{Tin, percent} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of the standard potassium iodate solution required to titrate the sample,

B = tin equivalent of the standard potassium iodate solution in g per ml, and

C = mass in g of the sample used.

5. DETERMINATION OF ANTIMONY BY POTASSIUM BROMATE TITRATION METHOD

5.1 Outline of the Method — The sample is decomposed by concentrated sulphuric acid and potassium bisulphate and a solution is made with water, hydrochloric acid and sodium sulphite. Arsenic in the solution is removed by vigorous boiling and the solution is then titrated with standard potassium bromate solution.

5.2 Reagents

5.2.1 *Concentrated Sulphuric Acid* — *see* **4.3.1**.

5.2.2 *Potassium Bisulphate* — solid.

5.2.3 *Concentrated Hydrochloric Acid* — *see* **4.3.3**.

5.2.4 *Methyl Orange Indicator* — Dissolve 0·1 g of methyl orange in 100 ml water.

5.2.5 *Sodium Sulphite* — solid.

5.2.6 *Standard Potassium Bromate Solution* (*0·01 N*) — Dissolve 0·278 3 g of the potassium bromate (previously dried at 105 to 110°C for one hour) in water and dilute to one litre in a volumetric flask.

5.3 Procedure

5.3.1 Transfer a suitable mass of the sample (so as to contain about 0·1 g of antimony) in a 500-ml round bottom flask. Carry out complete decomposition of the sample with potassium bisulphate and concentrated sulphuric acid as described in **4.4.1**. Cool, add 10 ml of water, 75 ml of concentrated hydrochloric acid and 1 g of sodium sulphite. Boil vigorously until the volume is reduced to 60 ± 5 ml (temperature should not exceed 110°C) to ensure removal of arsenic. Dilute to 300 ml, heat to 80-90°C. Titrate the solution at a temperature of 80-90°C with potassium bromate solution adding 3 drops of methyl orange as indicator, preferably added near the end of the titration. The end point is indicated by the disappearance of the red colour of the methyl orange. Carry out simultaneously a blank determination on the reagents.

5.4 Calculation

$$\text{Antimony, percent} = \frac{(A - B)\,C \times 0.0609 \times 100}{D}$$

where

A = volume in ml of the standard potassium bromate solution required to titrate the sample,

B = volume in ml of the standard potassium bromate required to titrate the blank,

C = normality of the standard potassium bromate solution, and

D = mass in g of the sample used.

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